

Effect of Soaking Time to the Bending Strength of Porcelain with Palm Oil Fuel Ash

Mohamad Zaky NOH^{1,a*}, Hassan Usman JAMO^{2,b}, Mohd Al Amin MUHAMAD NOR^{3,c} and Zainal Arifin AHMAD^{4,d}

¹Materials Physics Laboratory, Faculty of Science, Technology And Human Development, Universiti Tun Hussein Onn Malaysia, 86400 Batu Pahat, Johor, Malaysia.

²Department of Physics, Northwest University Kano, Nigeria.

³School of Fundamental Sciences, Universiti Malaysia Terengganu, 21300 Kuala Terengganu, Malaysia.

⁴School of Material and Mineral Resources Engineering, Universiti Sains Malaysia, 14300 Nibong Tebal, Penang, Malaysia.

^azaky@uthm.edu.my, ^bjamouhfce@gmail.com, ^cal_amin@umt.edu.my, ^dsrzainal@usm.my

ABSTRACT. Silica from a treated palm oil fuel ash (POFA) was used as a substitute material in producing an improved porcelain ceramics. POFA was grounded in a ball mill until the median particle size was reduced to about 50 μm . It was heated at 600 $^{\circ}\text{C}$ for 1.5 hours in an electric furnace. Then it was substituted quartz in porcelain composition from 5 wt.% up to 25 wt.%. The mixed powder was pressed into pellets at pressure of 91 MPa. All the pellets were sintered at 1100 $^{\circ}\text{C}$ for the soaking times of 1 hour, 2 hours and 3 hours, respectively. It clearly indicated that the bending strength of the samples increases with the increase in POFA substitution and the increase in soaking time. With a value of 45 MPa bending strength of the samples containing 15 wt.% of POFA and sintered at a soaking time of 2 hours is the highest. This could be attributed from the increase in mullite and sharp microstructural changes. Porcelain containing POFA has about 7% weight reduction compared to the standard porcelain.

Keywords: Bending strength, Bulk density, POFA, Porcelain, Quartz, Soaking time;

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1. INTRODUCTION

Porcelain ceramic is primarily composed of kaolin, feldspar and quartz, heat-treated to form a mixture of glass and crystalline phases (Na_2O , K_2O)– Al_2O_3 – SiO_2 [1]. Most of the reactions occurring during sintering are kinetically governed processes that do not reach thermodynamic equilibrium, since the industrial cycles are as short as 1 hour. Hence, it is very common for the finished product to contain crystals of quartz and feldspars that have not been entirely transformed. The plastic kaolin components, necessary to attain high green densities and advanced densification of the sintered product in a single fast sintering cycle, disappear completely to form mullite [2]. From an economic perspective, porcelain stoneware accounts for 33% of the entire ceramic tiles market world-wide, at sales levels of 119.5 Mm^2 in 1997 and 163.8 Mm^2 in 1998. Moreover, in the last decade the global production, inclusive of porcelain ceramics, was oriented mainly towards large formats (less than 25 tiles per m^2), increasing from 42% in 1988 to 82% in 1998 [3]. Despite the commercial interest in developing porcelain ceramic, very little research has been conducted in the field [4], leaving significant opportunities for investigation and study, particularly in the two topical areas of bending strength property and soaking time, which are the main topic of this paper. The objective of this

paper is to study the bending strength of porcelain ceramics by substitution of quartz by palm oil fuel ash at different soaking times.

2. MATERIALS AND METHODS

The removal of excess carbon and other unburned organic materials contained in POFA is important to avoid their potential negative effect on finished product. The untreated POFA was dried in an oven at 100 °C for 24 hours and then grounded in a ball mill to reduce the particle size to improve reactivity. Then it was sieved using a set of sieves to remove the particles coarser than 50 µm. The milling time was approximately 1.5 hours at 200 rev/min. The calcination process of the untreated POFA was done at 600 °C for 1.5 hours in an electric furnace.

Porcelain powder was grounded separately in a ball mill. The treated POFA was gradually incorporated into the porcelain powder from 5 wt.% to 25 wt.% as shown in Table 1. The mixing process was done for 1.5 hours in a ball mill. The mixed powder was pressed into pellets at mould pressure of 91 MPa. All the pellets were sintered at 1100 °C for the soaking times of 1 hour, 2 hours and 3 hours, at a heating rate of 5 °C/min, respectively. The chemical composition of the raw materials were studied using X-ray fluorescence (XRF) machine while the amorphous structure of the samples were identified through XRD, and the microstructural features were studied by SEM. The bending strength was determined. The formula for the bending strength of a beam in three-point is as follows.

$$S = \frac{3PL}{2bd^3} \text{ (MPa)} \quad (1)$$

where, P is break load (N), L is outer (support) span (N), b is specimen width (cm) and d is specimen thickness (cm).

3. RESULTS AND DISCUSSION

XRF analysis is proficient in analyzing material contents inside POFA, hence the amount of SiO₂ can be observed. The presence of various compounds within porcelain and POFA sample can be seen in Table 2. This table shows the result of XRF analysis of porcelain and POFA. It is evident that SiO₂ is the major composition in all the raw materials viz: POFA, kaolin, feldspar and quartz with 66.91 wt.%, 69.30 wt.%, 72.70 wt.% and 99.40 wt.% respectively. Followed by alumina with 6.44 wt.%, 24.30 wt.%, 16.40 wt.% and 0.22 wt.% respectively.

Table 1 The composition of quartz substitute by POFA (wt.%)

Mix Number	Kaolin	Feldspar	Quartz	POFA
AF1	50	25	25	0
AF2	50	25	20	5
AF3	50	25	15	10
AF4	50	25	10	15
AF5	50	25	5	20
AF6	50	25	0	25

Table 2 Chemical analysis of POFA and porcelain

Sample	Content (wt.%)												
Composition	SiO ₂	Al ₂ O ₃	FeO ₃	CaO	K ₂ O	P ₂ O ₅	MgO	SO ₃	Na ₂ O	MnO	TiO ₂	CO ₂	LOI
POFA	66.91	6.44	5.72	5.56	5.20	3.72	3.13	0.33	0.19	-	-	-	2.30
Kaolin	69.30	24.30	0.27	-	2.44	-	-	-	-	-	0.27	0.10	0.36

Feldspar	72.70	16.40	0.40	0.50	2.42	-	-	-	6.87	0.29	-	0.10	0.32
Quartz	99.40	0.22	-	-	-	-	-	-	-	-	-	0.10	0.28

Fig. 1 shows the graph of porosity versus POFA content, it can be seen from the graph that the least porosity was recorded with a value of 3.8%, 2.4% and 3.0% at a soaking time of 1 hour, 2 hours and 3 hours, respectively on 15 wt.% of POFA. As the substitution of quartz by POFA increases the porosity decreases until it reaches a minimum value and then increases again. Meanwhile the soaking time of 2 hours shows the least porosity at all composition of substituted POFA.

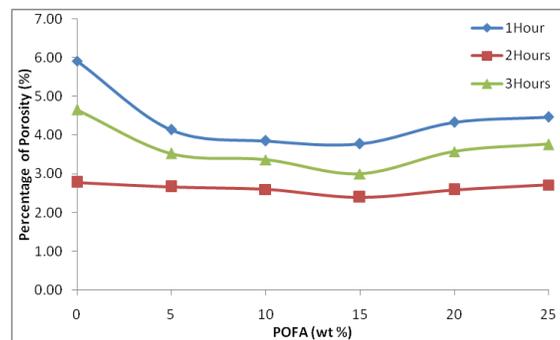


Fig. 1 Effect of soaking time on the percentage of porosity

The sintering process is mainly dominated by surface diffusion at low soaking time and by volume diffusion at high soaking time [5-6]. According to Abadir et al. [7] the temperature coupled with the soaking time have a major role in increasing the diffusion coefficient. Therefore, an increase in soaking time can promote the densification process. In addition, the existence of oxides, such as CaO and K₂O in POFA were beneficial to the formation of low melting liquid. With the increase of soaking time, the low viscosity liquid flowed, filled the pore and consequently helped the densification process. The density of the sintered samples decreased with the high amount of iron content. However, porosity decreases as the substitution increases beyond 15 wt%, this is as result of bloating as confirmed by the SEM (Fig. 4). This result is in agreement with the result obtained by Yanyi [8] and Noh et al. [9].

The bending strength of porcelain tiles were measured as function of soaking time (Fig. 2). It is obvious that these changes in bending strength are related to porosity and bulk density developments in the bodies. The highest bending strength was achieved with value of 31 MPa, 36 MPa and 34 MPa for 1 hour, 2 hours and 3 hours on 15 wt.% of POFA, respectively. The bending strength increased with an increase in soaking time and substitution of POFA. However, after reaching the maximum soaking time at 2 hours, the bending strength begins to decrease due to the porosity development [10].

The bending strength is strongly dependent on the microstructure, especially on defects such as pores and cracks. The bending resistance of porcelain is strictly correlated with the material porosity. The prepared porcelain tile with a high bending resistance presents a very compact texture, a smooth surface with low porosity (Fig. 4(b)). The surface with a wide range of pore sizes, in particular coarse, shows lower mechanical performances. In a study carried out by Ece and Nakagawa [11] proposed that porosity may affect the mechanical properties of ceramic materials in two ways. First, it reduces the effective cross-sectional (load-bearing) area such that the mechanical property will be dependent on the minimum contact-solid area. The minimum solid (load-bearing) area is the actual sintered or the bond area between particles in the case of stacked particles, and it is case of stacked bubbles. Second, porosity leads to stress concentrations near the pores such that under mechanical loading, the true stress in the material is higher near the pores than at a far distance from them [9]. The surface of porcelain presents diverse micro structural elements, either intrinsic

features of the ceramic body (e.g. residual pores) or superficial defects created during the grinding process. This result is in line with bulk density results shown in Fig. 1.

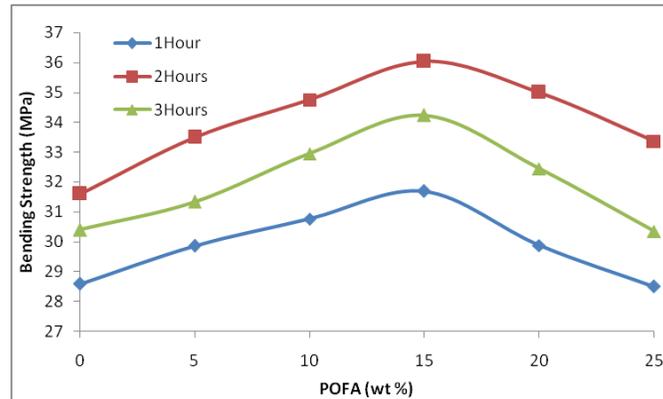


Fig. 2 Effect of soaking time on the bending strength

Fig. 3 shows the XRD patterns of the porcelain body after sintering at 1100 °C, between the soaking time of 1 hour and 3 hours. The peaks due to a given phase have been labelled. The main mineralogical phases of the sample are identifiable. They are: quartz (ICDD 046-1045), mullite (ICDD 089-2645) and cristobalite (ICDD 039-1425). The intensity of peaks resulting from quartz increases after sintering between the soaking time of 1 hour and 2 hours. Although the quartz is present at all sintering temperatures, the intensity of peaks decreases above the soaking time of 2 hours. Mullite and cristobalite phases, increases with increase in soaking time (Table 3). The reason for the increase in mullite is as result of reaction between Al₂O₃ and SiO₂ in the presence of temperature. While for cristobalite is as a result of quartz disilusion. This result is in agreement with the results obtained by Martín-Márquez et al. [12].

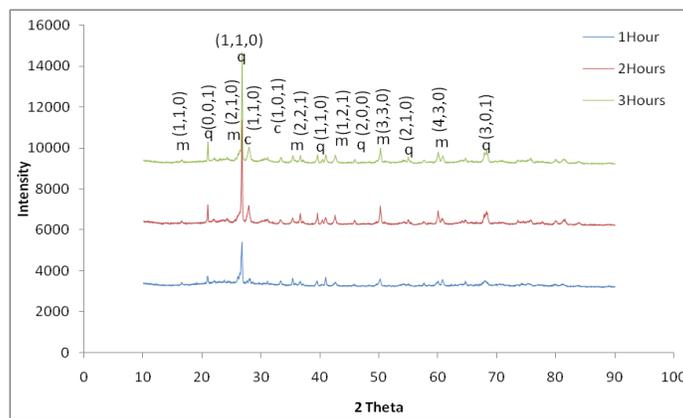


Fig. 3 The XRD curves of the samples containing 15 wt.% POFA

Table 3 XRD quantitative analysis of the body samples containing 15 wt.% POFA

Soaking time (Hour)	Quartz (%)	Mullite (%)	Cristobalite (%)	Glassy phase (%)
1	39.2	18.5	20.0	22.3
2	38.0	31.2	23.7	7.1
3	32.5	15.1	10.4	58.0

Fig. 4 shows SEM images of the body mixes sintered at different soaking times. The samples sintered at 1100 °C at a soaking time of 1 hour (Fig. 4a) shows a typical under sintering ceramic microstructure with high porosity. The general microstructure consists of quartz particles, kaolin-derived agglomerates, and a fine matrix of decomposed kaolin and feldspar.

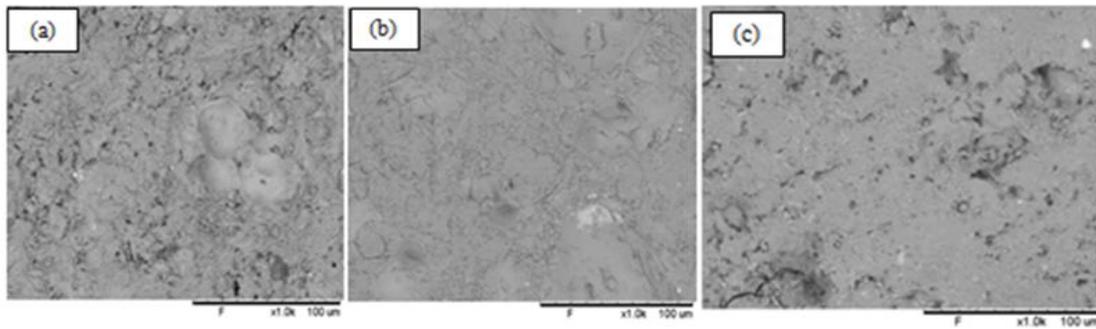


Fig. 4 SEM of the mixed samples containing 15 wt.% of POFA sintered at a soaking time of (a) 1 hour, (b) 2 hours and (c) 3 hours. All micrograph were taken with 1000X magnification

The microstructure of the sample sintered at a soaking time of 2 hours (Fig. 4b) is more compacted than that observed at lower soaking times because of the liquid phase formed from the fluxing agents and secondary mullite crystals. Although analysis allowed the quantification of 31.2% of mullite at the soaking time of 2 hours, its (mullite) formation is not visible by SEM, likely due to the small size of the first developed mullite crystals. At the soaking time of 3 hours (Fig. 4(c)) micro-cracks were developed this could be attributed to bloating due higher soaking time.

4. SUMMARY

The bending strength of porcelain body was found to increase with increase in substitution of quartz by POFA and soaking time. With a value of 36 MPa the highest bending strength was achieved at a soaking time of 2 hours on 15 wt.% substitution of quartz by POFA. Furthermore, the progressive substitution quartz by POFA in a porcelain body resulted in early vitrification of the mixtures. The bending strength result indicated that POFA has the potential to be used as a substitutive material and can improve the strength of the recycled aggregates porcelain.

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